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**FINAL REPORT
RESEARCH STUDY ON DEVELOPMENT OF
ENVIRONMENTAL FRIENDLY SPRAY-ON FOAM
INSULATION (SOFI) FOR THE EXTERNAL TANK (ET)**

**PREPARED FOR:
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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September 27, 1996

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13. ABSTRACT

The selection and qualification of four foams using a more environmental friendly HCFC-141b blowing agent replacing foams that used CFC-11 blowing agent for the ET LWT has been addressed along with problems and solutions that were encountered during verification. Effort on two lower density spray foams for the ET SLWT are presented, but predicted weight savings were not encouraging. Suggestions for possible problem solving are included along with a new approach for selecting foams for qualification as back-up foams for the foams used on the ET LWT. Investigated three resins for use as thermally sprayed coatings for corrosion prevention on metal. The best coating was obtained with a thermoplastic polyimide resin, and if it can be applied without adversely affecting the temper of aluminum, this coating has a good chance of meeting ET requirements. Possible third generation blowing agents have been shown usable in polyurethane spray and pour foams, and solubility in isocyanate foam components are acceptable. Considered aerogels as insulation materials on space vehicles, and suggested a liner for a LOX composite tank.

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FINAL REPORT**RESEARCH STUDY ON DEVELOPMENT OF
ENVIRONMENTAL FRIENDLY
SPRAY-ON FOAM INSULATION (SOFI)
FOR THE EXTERNAL TANK (ET)****SUMMARY**

This study addressed formulation and processing problems encountered with the Spray-on Foam Insulation for the External Tank, part of the Space Shuttle. The original foam insulation systems were mature, but used the freon CFC-11 as the blowing agent. This freon has a high ozone depletion potential and therefore U.S. suppliers were required to stop production by the end of 1995. The foam insulation industry chose HCFC-141b as its replacement. This mandated change while a drop-in for most of the industry required reformulation and processing changes to produce foam insulation having the physical properties required for External Tank mission profile. The known problems encountered on the four new foam insulation systems were defined, analyzed and resolved during this study. Insulation weight savings challenges were studied as were foam insulations using blowing agents with zero ozone depletion potential. Insulation systems for future use were evaluated and studied. Corrosion protection systems that also bond the insulation to the tank were studied as the current one uses chemicals on the list to be replaced in the future. Each of these major topics is summarized here with greater detail provided in the body of this report.

This study analyzed the problems associated with developing, qualifying and application of four new foam insulations to the ET used to provide quality cryogenic fuels to the SSME engines, to prevent ice/frost formation on the pad, and to protect the tank from radiant and aeroheating during ascent and re-entry. The Shuttle's External Tank Thermal Protection System (TPS) must be able to withstand cryostrains without cracking or debonding resulting from filling the LOX and LH₂ tank with cryogenic fuels. Additional strains are induced from pressurization of the tanks for flight. Two of the foam systems used on the ET had to be replaced with back-up foam systems while efforts were underway to replace the CFC-11 blowing agent with the more environmentally friendly HCFC-141b blowing agent. One foam was replaced because the manufacturer of the two primary polyols (used in the "B" component) discontinued production of those polyols. The other foam was replaced because the HCFC-141b blowing agent in the formulation did not produce foam meeting the ET specification. All four new foam systems using HCFC-141b blowing agent have been successfully applied to flight hardware.

Reducing hardware weight of the ET is the objective for the Super Light Weight Tank Project (SLWT). Weight reduction goals have been set for the aluminum tank and for the TPS. The current TPS goal is 250 pounds. The Lockheed Martin Company, the prime contractor for the ET, conducted development work on a lighter weight version for two of the new foam systems now being used on current flight hardware, called the Light Weight Tank (LWT). Based on test data for the lighter weight version of the two new foams, calculated weight savings were not sufficient to justify continued effort to qualify these foams for use on SLWT. TPS weight savings will apparently be limited to that saved by a much tighter control of applied spray foam thicknesses. Varying thicknesses of foam insulation on the plus Z side of the ET are required from increased heating rates due to protrusions and to the proximity of the Orbitor. Also, the SLWT Project would like to increase the maximum internal temperature for the ET's forward Ogive from 300°F to between 350-400°F. With the present new foam system this cannot be allowed. Two foams were examined for minimal testing that could possibly be used on the Ogive that would allow the higher internal temperature.

With the selection of the four new foams for the LWT, there are no qualified back-up replacement foam for any of the four foam systems. A new method for selecting foams for qualification as back-up foams was proposed.

Lockheed Martin has shown that third generation blowing agents (HFC & HFE), can be used as blowing agents in polyurethane spray foams and polyurethane pour foams. Test data indicate that fluorine substituted propanes and fluorine substituted methyl ethyl ethers have adequate solubility in polyisocyanate foam. Based on expected solubility of these blowing agents in the sprayed foams, the strain capabilities of these foams will be less than foams blown with CFC or HCFC.

Three resins were investigated for application to metal surfaces by a thermal spray process for corrosion protection. The most promising coating was obtained with a thermal plastic polyimide resin. If this polyimide resin can be thermally applied to aluminum without adversely affecting its temper, it would have a high potential of meeting ET requirements over the temperature range of -423 to +400°F.

The future availability of the ET primer is somewhat uncertain because of a change in ownership, a move of the production facility, and environmental constraints. A possible source of a modified epoxy resin that has potential for replacing the Super Koropon resin in the present ET primer, if it becomes unavailable, has been located.

Research has identified a new type of foam called aerogels. At this time they form porous molecular-scale foams and are formed using supercritical processing with pressures over 1000 psi and temperatures over 300°C. They can be made from silicates or formaldehydes. Aerogels have been considered as insulation material on Space Vehicles. The open cell structure of the aerogels prevents them from being used directly as insulation for liquid hydrogen tanks; hence no use is seen for aerogels on the ET LWT or SLWT. On reusable single stage to orbit vehicle, they could possibly be used to protect the isocyanurate foam insulation on cryogenic tanks.

The use of thermally sprayed FEP Teflon as a liner for LOX composite tank causes a concern for possible thermally induced cracking of the liner when the tank is filled with LOX due to the differences in coefficient of contraction. It was suggested that a liner could be made by using a DuPont film of Kapton H/FEP Teflon. This liner would be subject to the same differences in coefficient of contraction, but with the Kapton H film backing up the Teflon film, it is believed that this combination has a better chance of success due to the toughness of the Kapton H film.

The foam systems on the ET have been transitioned from using the freon (CFC-11) blowing agents which are more environmentally unfriendly to hydrochlorofluorocarbon blowing agents. This required both formulation and process changes to meet the stringent physical properties for the foam insulation on the ET. This study documents the major problems encountered in this transition and discusses replacement primers and some aspects of developing foams that are totally environmentally friendly.

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J. M. STUCKEY

I. ABSTRACT

The selection, qualification, and application of four foams using a more environmental friendly HCFC-141b blowing agent replacing foams that used CFC-11 blowing agent for the ET LWT has been addressed along with problems and solutions that have been encountered during verification and application on flight hardware. Efforts on lower density NCFI 24-124 and SS-1171 spray foams for the ET SLWT are presented, but after considering all test data the potential weight savings did not justify further effort to qualify these lighter weight foams. Weight savings from foam insulation on SLWT will be obtained by better control of the applied thickness during spray application. Suggestions for possible problem solving and production application are included along with a new approach for selecting foams for qualification as back-up foams for the ET. Investigated three resins for use as thermal sprayed coatings for corrosion prevention on stainless steel and aluminum. Test data confirmed that the thermoplastic polyimide resin was the most promising coating. Further effort is needed to confirm that the thermoplastic polyimide resin can be applied to aluminum surfaces without adversely affecting the temper of the metal. If this is possible, test data indicates that this coating will meet ET requirements over a temperature range of -423°F to +400°F.

Change in ownership of the manufacturer and environmental constraints have somewhat clouded the future for the ET primer. A source for a modified epoxy resin has been located that has promise for being a replacement of the Super Koropon resin used in the ET primer. The lack of a solvent for thermoplastic polyimide resin precludes its use in primer systems. Some potential third generation blowing agents have adequate solubility in the "B" component of polyurethane and polyisocyanate foams, but problems can be expected if the boiling points are lower or higher than 25 to 30°C. Lower solubility in the spray foams may lower the strain capabilities of the foams, but in turn may allow the internal temperature of the Ogive to increase to 350°F or possibly to 400°F. Current capabilities of aerogels precludes their use for insulation on LH₂ tanks. If aerogels with refractory coating can survive ascent and re-entry heating, they may find use on the proposed reusable vehicle of one stage to orbit. A Kapton H/FEP Teflon film from DuPont is proposed as a liner for a composite LOX tank.

II. INTRODUCTION

Early in the 1980's, it was obvious that the ozone layer in the upper atmosphere that protects the earth from some of the more severe rays from the sun was being depleted. Evidence pointed to the fact that CFC (chlorofluorocarbon) gases used in many commercial applications reacted and destroyed the ozone. Industry started developing replacement gases for the CFC that either did not react with the ozone (HFC-hydrofluorocarbon) or reacted at a much slower rate (HCFC-hydrochlorofluorocarbons).

All four foams used on the ET for thermal protection utilized CFC-11 as the blowing agent. HCFC-141b was developed by industry to replace temporarily the CFC-11 until economical procedures can be developed to produce a HFC for this use. Phase out of production of CFC-11 took place on December 31, 1995 in most areas of the world.

Preliminary studies had indicated that HCFC-141b would be a drop-in replacement for CFC-11. This did not turn out to be the case. When actual qualifications of the four ET foams with HCFC-141b blowing agent were initiated, problems were soon encountered. While trying to solve these problems it was learned that two of polyols used in "B" component of CPR-488 foam (about 50% of the "B" component) would no longer be produced after early 1995; hence the loss of CPR-488 foam used as barrel insulation for the LH₂ tank, LOX tank, and intertank.

Other problems that were addressed include the somewhat cloudy future of the epoxy based primer used on the ET, problems with composite fairings at higher heating rates, possible lower strain capabilities of foams that use third generation blowing agents, the potential of current aerogels as insulation on cryogenic tanks, and potential liners for graphite/epoxy composite LOX tanks.

III. FOAMS FOR ET LIGHT WEIGHT TANK (LWT)

A. NCFI 24-57 FOAM FOR ET AFT DOME LH₂ TANK

When the CFC-11 blowing agent in the NCFI 22-65 foam system now used on the aft dome of the LH₂ tank (ET LWT) was replaced with HCFC-141b, the foam system was renamed NCFI 24-57. Earlier tests have shown that this new foam system meets all the requirements for a foam to be used on the aft dome of the LH₂ tank.

For the final qualification of NCFI 24-57 foam for use on the aft dome of the LH₂ tank of the ET, a full scale spray foam application of this material to the aft dome of the LH₂ tank was required. For this test to pass, it required that all plug pull values have a minimum strength of 40 psi. The requirement as written did not allow for retest in adjacent areas. Foam applied to lead-in and lead-out panels were also required to have acceptable densities, and also pass cryoflex tests at -423°F.

1. Two verification sprays of the NCFI 24-57 on the aft dome of the ET resulted in two or three low plug pull values (below 40 psi) for each spray. Plug pulls adjacent to the low plug pulls gave acceptable values. On this basis the verification sprays are probably acceptable. The average strength of the plug pulls seem a little low for the bond tension data. However, it was noted that none of the plug pull knit line failures were above 50%.
2. Two additional verification sprays of the NCFI 24-57 foam on the aft dome of the ET were performed. On the first additional spray, one or two low plug pull valves (below 40 psi) were again encountered. Although additional plug pulls in the vicinity of the low plug pulls had valves above 40 psi, a fourth verification spray was run. In this test, all the plug pull valves were above 40 psi. At this time it was considered that the NCFI 24-57 foam was ready to be sprayed on flight hardware and the first flight hardware was sprayed. The foam on the tank looks good.

3. In preparation to confirm that the foam on the LH₂ tank aft dome met all requirements, most of the foam was accidentally removed from the lead-in test panel that was sprayed at the start of foam application. Acceptance of the foam on the tank requires certain tests be run on a lead-in test panel that is sprayed at the start of foam application and also on a lead-out panel sprayed at the termination of the spray application. These tests include cryoflex, density, bond and compression strength, etc. Since cryoflex and perhaps other tests cannot be run on the lead-in test panel sprayed at the initiation of foam application, part of the tests required to confirm that the foam on the tank meets requirements cannot be run. The foam on the aft dome of the LH₂ tank is probably acceptable, but all the tests required cannot be met. In my opinion, the tank should be stripped and resprayed especially since this is a new foam and used for the first time on flight hardware.

4. The aft dome of LH₂ tank for ET-80 was sprayed with foam NCFI 24-57 during the early part of February. The foam met all the requirements for acceptable flight quality foam. Since the NCFI 24-57 has now become a foam acceptable for use on the ET, it was dropped from the qualification program.

B. NCFI 24-124 FOAM FOR THE ET TO REPLACE CPR-488 FOAM

North Carolina Foam Industries was requested to supply a foam (NCFI 23-66) using HCFC-141b as the blowing agent, replacing CFC-11. NCFI 23-66 (using CFC-11 as the blowing agent) had previously been qualified as a foam for use on the ET as alternate foam for CPR-488. This new foam was designated as NCFI 24-124.

1. The as received NCFI 24-124 (Lot #1) had a barrel spray density of 2.45 lbs/ft³, and generally had higher tensile strengths than the NCFI 23-66 data base (DB) and appears acceptable for use on the Ogive.

2. The NCFI 24-124 adjusted with 2.5% HCFC-141b had a lower density than NCFI 23-66 DB (2.15 vs 2.07 lbs/ft³ barrel sprays) and also gave foams with lower tensile strengths than the DB at room temperature, +200°F, and +300°F. An Ogive spray of this material gave lower tensile strengths than NCFI 23-66 DB. At 300°F the 20 psi tensile strength showed that this foam would be unacceptable for use on the Ogive.

3. Barrel sprays of NCFI 24-124 (2.45 lbs/ft³) and 1% adjustment with HCFC-141b (2.27 lbs/ft³) gave foams with about the same tensile strengths over the temperature range of -423°F to +300°F.

4. LMC/MAF also obtained new quantities of NCFI 23-66 (CFC-11 blowing agent). Although the new NCFI 23-66 had a higher density than the DB (barrel spray density of 2.16 vs. 2.27 lbs/ft³), it had lower tensile strength at room temperature, 200°F and 300°F, than the DB. With the low strength values at 300°F, this foam would probably be unacceptable for use on the Ogive. No additional effort is planned on this material until other more pressing problems have been resolved.

5. Test in the Hot Gas Facility showed that NCFI 24-124 adjusted with 2.5% HCFC-141b had somewhat higher recession rates than the foams made with "as received" NCFI 24-124. However, the recession rates for the 2.5% adjusted foam was still lower than the recession rates for CPR-488 foam in the same test facility.

6. Based on tensile and other tests LMC was instructed to concentrate their efforts for qualifying NCFI 24-124 with 1% adjustment of HCFC-141b blowing agent for ET LWT rather than also working on an optimized density version of NCFI 24-124 foam for the ET SLWT. The NCFI 24-124 adjusted with 1% HCFC-141b was qualified as a replacement foam for CPR-488 for use on the ET LWT in the April/May time frame of 1996.

7. LMC added 1% of HCFC-141b to Lot #2 of NCFI 24-124 at MAF. Information developed on this foam system was presented at a meeting at MSFC on January 10, 1995. Bond tension and compression strength properties appeared to be adequate, but tensile values are generally 6 psi or more lower than those of CPR-488 foam, except at -320°F, the density was somewhat higher (2.28 lbs/ft³ vs. 2.14 lbs/ft³). At +300°F the bond strengths were 5 psi lower than that for CPR-488 (31.2 psi vs. 36.9 psi) indicating possibly a lower factor of safety on the Ogive.

8. Cryoflex tests at 61 KSI showed 100% passing; but at 65 psi only 92% passed. The tests at 65 psi are definitely an over test. Wind tunnel tests showed lower recession rates than those encountered with CPR-488. New monostain data at -423°F indicates a very slightly lower strain capability than reported in the first bi-monthly report. The strain to failure in these tests showed a failure strain (in/in) of 0.0178 compared to the earlier data (0.0185) which is lower than that of CPR-488 (0.0211). This very slight drop in strain capabilities is not important as the ET does not see strains to this level.

9. After Lot #2, all future lots of NCFI 24-124 foam had 1% additional HCFC-141b blowing agent blended into this foam at NCFI plant. Although more blowing agent was added, there was no change in the name of the foam; hence causing some confusion.

10. The new NCFI 24-124 (Lots #3 and subsequent Lots) have been successfully sprayed duplicating the equivalent of barrel and Ogive sprays for the ET. Densities were generally 2.24 to 2.30 lbs/ft³. Adequate bond tension and FWT strength were demonstrated over the temperature range of -423°F to +300°F. Wind tunnel tests have shown lower recession rates for this foam than were obtained with CPR-488 foam.

11. Back face temperature tests have shown that this foam meets the requirement of passing a 300°F back face temperature under vacuum conditions without a failure. The NCFI 24-124 plus 1% HCFC-141b foam failed at 320-330°F range while CPR-488 fails around 375°F.

12. The new monostrain tests have a few questionable test results but still appear adequate. The initial monostrain tests (3) had a failure strain of 0.0172 in/in at -423°F. With 29 additional tests the average failure strain had increased to 0.0202 in/in at -423°F. This same foam had a failure strain at -320°F of only 0.0179 in/in (4 tests). It would be expected that the foam at -320°F would have a higher failure strain than it would have at -423°F. However, with more test data the strain data at -320°F might exceed that at -423°F.

13. Cryoflex data on this foam at -423°F showed 100% passing at 61 KSI and only 8% failure rate at 65 KSI (109 test). Gradient cryoflex testing on 100" and 50" radius anvils did not give consistent results. The first 2 tests on foam 1.22" thick failed at 50 KSI on the 100" radius anvil. The last 2 tests on foam 1.32" thick passed both the 100" and 50" radius anvils when tested at 65 KSI.

14. Thermal conductivity tests at MAF had indicated that NCFI 24-124 with 1% additional HCFC-141b foams has a little higher thermal conductivity than CPR-488 foam. Thermal data in the -423°F to -250°F range was needed to better calculate the affect that this higher thermal conductivity may have on the probability of ice/frost formation on the pad. It was edicted that there will be no increase in foam thickness on the ET. To quantify the effect that this higher thermal conductivity of the NCFI 24-124 foam may have on actual surface temperature, a flat tank with CPR-488 on one side and NCFI 24-124 foam on the other side (both trimmed to the same thickness), was tested at MSFC, and showed about the same thermal conductivity for both foams. Guarded hot plate data from Halometrix showed essentially the same thermal conductivity data for old CPR-488 foam (1987 or older), new CPR-488 foam, and NCFI 24-124 foam. This was surprising particularly for the old and new CPR-488 foams as foam insulation tends to lose CFC-11 from inside the cells of the foam which is replaced with air giving a higher thermal conductivity for the older foam. Based on the above data, LMC considered NCFI 24-124 as viable drop-in replacement for CPR-488. The proposed update for nominal and maximum design thermal conductivity for ET sidewall foam material does not impact LWT and SLWT thermal design.

15. NCFI 24-124 foam passed all qualification tests for replacing CPR-488 foam as the TPS material for the barrel sections of the LH_2 and LOX tanks, for the Ogive and for the intertank. Wind tunnel tests have shown lower recession rates for the NCFI 24-124 foam than for the CPR-488 foam. Combined environment tank test passed all requirements, and back face, vacuum heating tests exceeded the requirement of no divoting at 300°F . Mechanical properties are acceptable over the temperature range of -423°F to 300°F , and this foam has essentially the same thermal conductivity as CPR-488 foam; hence is considered a drop-in replacement for CPR-488 foam. Thermal strain requirements are acceptable as cryoflex tests at 61 KSI showed 100% passing and monostrain data is more than adequate.

16. A new problem surfaced with regard to long duration sprays. As the spray time exceeds 10 to 15 minutes, pressure tends to build up in the tip and later the spray pattern goes off. This has occurred with the modified Binks gun and the new Peter Allen TPV gun. To determine whether it is an equipment or a material problem, they sprayed this material with the same Binks gun used to qualify NCFI 23-66 for long duration sprays (LH₂ tank barrel spray) and encountered the same pressure build-up problem. Analysis on the resin removed from the internal area of the gun between the mixing chamber and the tip showed that the resin is a polyurethane. This suggested that one of the polyols may be contaminated with a more reactive polyol or that the catalyst activity has increased. Three possible solutions were considered to solve this problem; a. shorten the distance between the mix chamber and tip in the gun, b. change the orifice plate, and c. to drop the component temperatures from 135°F to either 100°F to 115°F. All of these showed some promise for eliminating or decreasing resin/varnish build-up in the gun. When sprays were made at MSFC using lower component temperatures, there was no evidence of a secondary rise from the trimerization reaction causing lower bond strength to the substrate or foam-to-foam bonds. Irregardless, I believe the trimer content may be affected. No solution to the problem has been found - more guns are used. SLWT spray appears to have less pressure build-up - no explanation.

17. This foam has passed all qualification tests and has been accepted as a qualified foam to replace CPR-488 foam on the ET. NCFI 24-124 has been successfully applied as the foam insulation on the cylindrical section of the LH₂ tank on ET-85 and to LOX and Intertank on ET-86.

18. In spraying the cylindrical part of the LH₂ tank, it was noticed that there was a mist in the sight glass. Later a fine white solid was found on the filter. This contamination came from the tank where the "B" component is blended, and is an acrylonitrile residue left in the tank from a previous lot of material at NCFI. This contamination has always been present, but was only detected after changing to the Variable Output Pumping System (VOPS) that has better filter than used on the H-IV units. There is apparently no problem associated with this contamination.

19. In spraying the LOX tank, problems were encountered on the Ogive. This foam failed to meet strength requirements when tested, and the foam was stripped and resprayed. It was assumed that this was probably caused by a deterioration of the spray pattern. This is not a new problem as it has occurred with CPR-488 foam. If the frequency of this problem increases with the NCFI 24-124 foam, I would suggest switching to the back-up gun when you approach spraying the Ogive. This would probably prevent stripping foam from the Ogive, and respraying.

20. With the event of thick/thin foam on the LH₂ tank, the question arose on where to place the panel for cryoflex specimen to evaluate whether the foam on the tank is acceptable or not. I believe it should be placed on the thin side (-Z side of tank) as this will be a more severe cryoflex test (higher density, closer knit lines).

21. The VOPS spray equipment has the ability to spray foam to various thicknesses that are really needed on the ET. ET-88 was sprayed using the VOPS trying to have a tighter control on the thickness of foam applied on the LH₂ tank. The initial effort on measuring thickness encountered some problem from the selection of transition areas when making the measurements. Two other thickness measurements were made with more careful selection of the area to make the measurement, and the result showed the thickness to be generally within new specification except in a few areas where the thickness was 0.01 to 0.02 inches above that required. With the ability to control the thickness of the foam in various areas to that required, TPS weight can be saved. This spray demonstrated the ability of the VOPS spray system to apply foam in various areas on the ET to specified minimum and maximum thicknesses during spray application. This ability to spray foam to desired thicknesses is very important to SLWT as this control of TPS thickness is a part of the overall weight reduction.

C. REPLACEMENT FOR BX-250 (CFC-11)

1. Current BX-250/HCF-141b produced unacceptable foam when sprayed at ambient temperatures at 65°F to 75°F (lower end of the processing box). At ambient temperatures of 80°F and above, acceptable foam is formed. The proposed primary replacement, SS-1171/HCF-141b appears acceptable except the density appears to be higher (2.3 lbs/ft³ BX-250, 2.46 lbs/ft³ SS-1171 Lot #3, 2.48 lbs/ft³ SS-1171 Lot #4). Based on those densities there would be a 40-50 lb. weight increase to the ET which is undesirable, especially for the super light weight tank.

2. The test data on these two lots of SS-1171 material, shows generally a strength decrease as compared to BX-250/CFC-11. Tensile tests, although acceptable, generally exhibit at least 50% and higher knit line failures. Tests have not resolved the high percentage of knit line failures.
3. BX-255/HCFC-141b, a minor reformulation of BX-250, encountered problems when stored with the stability of the flame retardant; hence it has been dropped from the program.
4. BX-260/HCFC-141b, a major reformulation of BX-250, appears to be about equivalent to BX-250/CFC-11 with respect to density and strength properties of the foam. The knit line failures in tensile tests are generally below 50%, which is desirable.
5. The test data for both the SS-1171 and BX-260 show an increase in densities when sprayed at ambient temperatures of 90°F and 100°F. This was unexpected, and has not been explained.
6. SS-1171 (HCFC-141b blowing agent) was selected as the foam to replace BX-250 (CFC-11 blowing agent).

D. SS-1171 (HCFC-141b) FOAM QUALIFICATION

1. LMC has concluded that this change to SS-1171 (HCFC-141b) foam does not cause a weight increase. The data shows that with 0-knit lines the SS-1171 has a lower density than BX-250, and with 1 knit line the two foams have about the same density. With more than 1 knit line BX-250 foam has a lower density.
2. A cure time study was run on the effect of longer cures on strength values of plug pulls. This study included testing after 24 hours, 72 hours, and two weeks. Of 16 samples from two panels, only 1 plug pull value after 24 hour cure exceeded the plug pull strength after 2 weeks of cure. On this basis it appears safe to assume that the strength of the foam will increase over that shown by 24 hour plug pulls.
3. An overlap time study showed no drastic fall off of properties when the overlap time was varied from 15 sec. to 75 sec.
4. The minimum room temperature for spray application of the foam was increased from 65°F to 70°F. This was done because of the heavier knit lines and rind at 65°F, and to add to the margin of safety.

5. To qualify SS-1171 (141b) foam as a replacement for BX-250, it has to pass the LOX Pal Ramp Test. In this case the SS-1171 foam was applied over CPR-488. On warm-up from the first cryogenic test delamination occurred in the second and third knit line of the CPR-488 foam. This was a very unusual failure mode. MAF concluded that the failure was due to some processing difficulties when it was fabricated. I believe that there must have been some difference in the warm-up procedure that caused the substrate to warm-up faster which would cause extra strain in the area where failure occurred. Later SS-1171 foam was applied over NCFI 24-124 (CPR-488 replacement) and when this configuration was tested, no problems were encountered and the test passed.
6. For the 2 validation sprays in cell "B" for the LH₂ tank forward dome on ET-85, the spray of SS-1171 looked good. Densities on the lead-in and lead-out panels were 2.4 to 2.5 lbs/ft³ which agrees with BX-250 data base. All other tests were acceptable.
7. In spraying the aft bulkhead of ET-89 LOX tank, rather rough appearing foam was obtained. On investigation it was found that the component temperature was 120°F to 125°F. The new specification requires the component temperatures to be 135± 5°F. The foam was stripped, and new foam was applied using component temperatures around 135°F. Later the component temperature requirement was changed to 135°F to 150°F, preferred 145°F to 150°F. The higher component temperatures have not eliminated the heavy knit lines, but have reduced the tendency on tensile tests for the foam to fail in the knit lines.
8. In completing validation spray, MAF found that on longer duration sprays on simulated flight hardware that better results were obtained using the H-11 rather than the FF pumping system. Some 7 pumping systems have been modified for production sprays.

9. The last four lots of SS-1171 have not met the tensile and compressive strength requirement after a 24 hour cure that were established from several lots of material that were used in the qualification effort. The compressive strength is low [around 19-20psi - (specification calls for a minimum of 24psi - on development/qualification lots - 30 psi)]. With the first of these lots only a slight improvement in strength properties were obtained when the cure time was extended from 24 hours to two weeks. The remaining material in this lot was surplused. For the second of these lots extending the cure to 48 hours increased the strength values above the minimum that is required. The density of this lot of foam is a little low. This low density was probably caused by the slightly higher concentration of blowing agent in the "B" component. The third lot also had low strength values that did not improve much on extending the cure. It was thought that processing problem might be the cause of the low strength values. Changes in processing did not appreciably increase the strength values. Finger printing has not been able to detect the cause of the low strength values.

All of these low strength foams suggest that the catalyst activity may possibly be reduced. If this is the case, then the shelf life of the foams will probably be reduced. MAF is sending samples of these foams to LMC/MSFC to see if they can determine if the catalyst activity has changed.

It seems that the material made for qualifying this foam is a little more reactive than production lots of the foam. Statistics seems to indicate that in most cases a 72 hour cure will increase the strength sufficiently to meet requirements. On this basis MAF has proposed lowering the requirements for the RAP tests so that a 24 hour cure will give foam strong enough for the lower requirement. Will MAF continue to monitor the foam for 72 hour cure and/or 168 hours? This suggestion for lowering strength requirement was not to well received by MSFC. LMC is still working this problem.

E. QUALIFICATION OF PDL 1034 POUR FOAM

1. PDL 1034 foam is the same foam as PDL 4034 that is now being used on the ET LWT except that the blowing agent has been changed from CFC-11 (PDL 4034) to HCFC-141b (PDL 1034). After demonstrating the properties of the two foams are essentially the same, qualification would be accepted when 2 verification pours for several selected areas were accomplished. Verification of flight pours are essentially complete. There efforts have shown that with PDL 1034 it is necessary to increase the size of the charge about 7%. Engineering for the verification are using weighed amounts of the "A" and "B" components rather than volume measurements for the components. On rough foam surfaces it may be necessary to smooth off the substrate to get 100% bond of the pour foam to the substrate. A bonus for using the PDL 1034 pour foam has occurred in some areas. They have been able to complete the fabrication by using a single pour in place of two or more pours that have been used when foaming with PDL-4034.

2. MAF presented a back-up plan to MSFC for the production mold used for the close-out for the LO₂ outboard support arm. This back-up plan will be used if acceptable pours can not be made with this mold. It is scheduled for delivery on 7/31/96, and is planned to be used on flight hardware in October 1996. The delivery of the mold slipped, and LMC presented a back-up plan.

3. The other new molds for flight hardware are essentially just duplication of the present molds with only slight modification. No pours are planned for these molds, but they will be carefully inspected.

4. It was learned that Urethane Technology has purchased PDL. Not only do we have a new vendor, but the two chemists that LMC has worked with to obtain good PDL-4034 (now PDL-1034) pour foam have also gone. This creates a situation that could lead to problems in obtaining future supplies of good PDL-1034. This is critical since this foam is also used by Rockwell and USBI, with LMC taking the lead. On a telecon LMC/MAF presented orally a plan for recertifying the PDL-1034 foam from the new vendor that seemed to be acceptable, but a more detail plan is needed. The first lot of foam from the new vendor was unacceptable. LMC is still working this problem.

F. TRAINING PROGRAM AT MAF FOR KSC PERSONNEL

1. LMC/MAF conducted a training program at MAF for KSC personnel on spraying SS-1171 (HCFC-141b blowing agent) and on pouring PDL 1034 (also using HCFC-141b blowing agent). All trainees did not pass on the first time, but the program has provided KSC with enough trained operators that all foam application can be done. I think the training program was needed, and I am sure there will be a pay off from smoother operations at the Cape.
2. KSC personnel thinks that they will not need the modified H-11 pumping system for spraying the third hard point, and believe that FF pumping system will be adequate.

G. PROBLEMS ENCOUNTERED WITH ET LWT FOAMS USING HCFC-141b AS THE BLOWING AGENT

1. CRUSHED NCFI 24-124 FOAM ON FLIGHT HARDWARE

- a. LMC/MAF has encountered a problem with crushed NCFI 24-124 foam on flight hardware. The crushed foam has occurred along the LOX feedline and in other high traffic work areas. This problem has been attributed to the lower compressive strength of NCFI 24-124 foam as compared to CPR-488 foam. Carelessness also has been a contributing factor based on the number of crushed foam areas on ET-85, ET-86 and ET-87. ET-86 has far fewer crushed foam areas than ET-85, and ET-87 has even fewer areas than ET-86. To help protect the foam, LMC will use a mat that distributes loads better.
- b. There is some concerns that undetected crushed foam areas could cause problems during reentry resulting in the break up of the ET at higher altitudes than desired. No problems are expected from crushed foam during ascent. Visual inspection will not detect consistently areas of crushed foam much below 20% crushed foam. Hands on inspection does not greatly extend the capabilities of detecting crush foam over that of close visual inspection. Therefore, areas of 10% or less crushed foam will probably not be detected. LMC thermal would like to have more wind tunnel tests to have a better understanding on how serious a problem that undetected crushed foam may cause.

c. LMC will repair all detected crushed foam areas. On ET-85 most of crushed foam areas were repaired simply by sanding off damaged foam leaving foam with a thickness that still exceeded the minimum required thickness in that specific area of the ET. When the SLWT tank replaces the LWT, sanding to remove crushed foam probably will not be a way to repair the damaged foam in most areas because of the tighter controls on applied foam thickness. When the sanded foam thickness is below minimum required thickness, additional good foam is removed to a minimum depth of 0.5 inch, and then PDL-1034 pour foam has been used to repair areas of around 40 in² on ET-85. I am concerned if the repair areas are allowed to continue to grow to longer areas.

2. LOW STRENGTH SS-1171 FOAM

a. Production lots of SS-1171 foam appear to be having trouble meeting the strength requirements that were established from data obtained on qualification lots of this foam after a 24 hour cure. If the cure is extended to 48 hours, 72 hours, 1 week and 2 weeks, the strength properties generally increase, and in most cases meet strength requirements. It is not clear how long the foam from the qualification lots was allowed to cure before the strength properties were determined. It was also indicated that some values may have been obtained in some cases on foam samples that have been to close to the edge of the panel. This could explain decreases in strength on longer cures for some lots of foam. The data on this foam appears inconsistent and no clear pattern is evident. However, the overall results tends to indicate that the qualification lots of foam are somewhat stronger than the production lots of this foam.

3. FAILURE OF PDL-1034 TO MEET STM REQUIREMENTS

a. The first production lot of PDL-1034 from Urethane Technology (new owner) made after the loss of the quality control engineer and the chemist technical manager did not meet all receiving inspection requirements. The foam had a slightly low cup density [2.1 vs. 2.2 pcf (min.)] and also a low compression strength [29 vs. 30 psi (min.)]. Although within specification the water content was at maximum of 0.6% and blowing agent was near maximum - 19 vs. 20%. A decision was made to return this kit to the vendor and a minimum audit was conducted. The audit was not too successful. LMC is still working the problem, and appears to be having some success.

H. ALTERNATE FOAMS FOR THE ET

1. PRESENT SITUATION

a. LMC has no alternate foam for any of the 4 foams used as TPS on the ET. Loss of any of the four foams for any reason would drastically affect LMC ability to continue ET production. LMC doesn't even have promising foams that they could start a qualification effort if required. Plant problems such as fires or strikes might be resolved by transferring production to another plant, but even this causes problems. Loss of a raw material used in "B" component could eliminate the foam system entirely.

2. PROPOSED FUTURE EFFORT ON BACK-UP FOAMS FOR THE ET

a. I do not propose sampling the industry for new foams that will meet the requirements for foams to replace the current foams now in use. If possible, I propose to have LMC (Lauri Rando) prepare a list of all materials used in each foam system now in use on the ET, back-up foams now being considered, and any foam under development at MAF/MSFC to determine how many of the foam systems use duplicate material. If there is duplication of material components then the loss of a single material could result in the loss of more than one foam system. This would show where the most effort is needed.

b. NCFI is critical since the NCFI 24-57 (aft dome foam) and NCFI 24-124 (side wall foam) use the same components. Assuming the polyols are considered the most critical component, then MAF should work with NCFI to come up with replacements for the polyols. A preliminary qualification program should be run for each of the polyols and later combining all the new polyols in a single system. At that time a decision could be made on the new foam to be completely qualified. LMC/MSFC should be heavily involved in this effort with the idea that duplication of effort should be avoided.

3. LMC/MSFC EFFORT ON ALTERNATE FOAM

a. LMC/MSFC has come up with an excellent idea for obtaining alternate foams for the ET that has a much greater chance of producing positive results than using the old method of canvassing the industry to see if they have any foam or foams that will meet ET requirements. LMC is working with the vendor for NCFI 24-57 and 24-124 foams (North Carolina Foam Industries) to come up with alternate materials for the present materials in their foam formulation. Data will be obtained on how effective the new materials are as compared to the present materials, and will also give leads to properties that are needed to improve performance. Using the old method it is not known how many of the same materials are included in both foam formulations (current and back-up foams). If materials are duplicated, then the loss of a single material could wipe out both the presently used foam and its back-up foam.

b. LMC has already looked at surfactants, and have come up with 3 new surfactants that look promising for replacing the presently used surfactant. They have also looked at changing the ratio of components in both the catalyst and flame retardant packages. No effort has been made to date on coming up with replacements for the two polyols in the "B" component. Since this is a joint project between vendor and LMC, a good relationship exists.

IV. FOAMS FOR ET SUPER LIGHT WEIGHT TANK (SLWT)

LMC is under contract with MSFC to build a much lighter weight ET as more payload is needed for future flights. On this basis they have looked at reducing the weight of TPS mainly by reducing density.

A. LOWER DENSITY NCFI 24-124 FOAM

1. In the initial effort to select a foam to replace the CPR-488 foam on the ET, 1% HCFC 141b blowing agent adjustment to the NCFI 24-124 foam gave a spray foam material with a density of around 2.25 lbs/ft², and based on properties it was selected as the foam for the ET LWT. At the same time data was obtained on a foam that had been adjusted with 2.5% HCFC blowing agent (1.5% more HCFC-141b than the NCFI 24-124 foam selected for the ET LWT). This foam had a spray density of around 2.10 to 2.15 lbs/ft³, and would give the desired weight reduction needed by ET SLWT. Unfortunately, strength properties were not adequate to meet the SLWT requirements. At that time it was decided that MAF should concentrate their efforts on qualifying the NCFI 24-124 for use on ET LWT. LMC/MSFC was given the job of modifying the NCFI 24-124 foam to give a lower density material but still having adequate strength to meet the SLWT requirements.
2. Early efforts included tests of seven surfactants, addition of small quantities of PF 5050 (a blowing and nucleating agent), and higher levels of triminization catalyst. Three of the surfactants, including the current one, were acceptable, and based on data it was elected to keep the current one. PF 5050 has limited solubility in polyol and isocyanate components, and it offered little or no improvement in the cellular structure of sprayed foam. Higher levels of triminization catalyst improved the reactivity profile but no increase in strength was noted in sprayed foam.
3. NCFI foams have 3 flame retardants - 2 non-reactive and 1 reactive. A test program was run varying both the quantity of each flame retardant in a particular spray and also the overall level of flame retardants (6.5 - 25% of polyol component). Test panels of each of these variations of flame retardants were sprayed at low output. The sprayed foams were evaluated by determining mechanical properties at -320°F, 70°F, and 300°F. Thermal data considered hot-gas recession and oxygen index. This data indicated that the current flame retardant package is near optimum.
4. Good results were obtained when LMC/MSFC added 0.5% water to NCFI 24-124 foam that is being qualified for use on the ET LWT. The resulting foam had a density of 2.17 lbs/ft³, and the strength properties were much improved over the control (NCFI 24-124 plus 1.5% HCFC-141b, density around 2.10 to 2.15 lbs/ft³). When the NCFI 24-124 foam with 0.5% added water was sprayed at 40 lb/min rate, the resulting foam had a density of around 2.05 to 2.10 lbs/ft³.

5. To summarize all these efforts on developing a light weight NCFI 24-124 foam, seven systems were sprayed at 40 lb/min rate. The first system was the NCFI 24-124 that is qualified for use on LWT ET. The second system had 1 1/2% more HCFC 141b added to the NCFI 24-124 foam. The third and fourth systems had 0.5% and 0.75% water added to the NCFI 24-124 foam. System five was NCFI 24-124 foam with adjusted flame retardant. System six was system five with 0.5% added water. System seven had reduced flame retardant in the formulation.

6. Test data obtained at MAF caused concern as their test data, particularly at room temperature, was consistently lower than that measured by LMC/MSFC. The lower test values usually had failures at or adjacent to the bond of the sample to the test fixture. It appears as if the lower test data can be attributed to a not fully cured adhesive. Data available tends to indicate that system 2, 5 and 6 and possibly 3 have some potential of meeting SLWT requirements.

7. In another effort to save weight on the SLWT, they are considering spraying foam on the -Z side to a thickness of 0.5 to 0.6 inches thick. Cryoflex tests on this thin foam passed. In other cryoflex tests on Al-Li, the NCFI 24-124 foam was still bonded to the metal after the metal substrate failed.

8. Weight pick-up from ice may off-set weight gained by spraying thin foam on the -Z side. Tests on the flat tank, with foam 0.5 inches thick, had a surface temperature about 70°F below ambient temperature.

9. Based on the most recent tests, the adjusted flame retardant system 5 was recommended for consideration as the foam to be used on LH₂, LOX, and intertanks on the SLWT ET. Bond tension data at R.T. and 300°F are essentially the same as that of NCFI 24-124 that has been qualified to use on LWT ET. However, at -423°F the bond strength of NCFI 24-124 foam is 12 psi stronger. The failure mode at -423°F is usually at the foam bond to the test block for the light weight NCFI 24-124 foam. If a better bond was obtained, it is believed that the strength of the light weight material would improve. The density of the light weight foam is around 2.1 lbs/ft³ as compared to around 2.3 lbs/ft³ for the regular NCFI 24-124 foam. Thermal conductivity appears to be lower and closed cell content higher for the light weight foam. Hot gas tests show a recession rate for the light weight foam to be about 30% higher than regular NCFI 24-124, but still below the -2 sigma curve for the CPR-488 foam.

10. Due to shortage of Al-Li metal, cryoflex specimens are using 7075-T₆ aluminum and need to pass 73 KSI when tested at a 500⁰ radius. Tests at 76 KSI and 78 KSI have shown for the failed specimen that the crack initiated in the ramp area and had about one inch debond from the substrate for both the regular and light weight NCFI 24-124 foams. About 95% of the cryoflex failure occurred in this way, and failures in this mode are usually classified as no test. LMC stress has recommended that the specimen be reinforced in the ramp area by bonding a scrim cloth with a RTV. Reinforcement in the ramp area eliminated cracks in this area, and cryoflex specimen passed.

11. Some people at LMC/MAF believed that the cracks occurred in the cryoflex tests due to the presence of liquid air. I suggested the cryoflex test be run in an atmosphere of gaseous helium. This was not too well received at MAF. I believe combined environment panels should be run for testing both the regular and light weight NCFI 24-124 to insure that these foams can survive the strains expected to be encountered on the SLWT. In my opinion acoustics and heat could be eliminated in this test initially because the adequacy of the foam to survive the stress loads is the major concern at this time. With the elimination of heat and acoustic, the test probably could be run at MSFC.

12. The adjusted flame retardant system 5 (Lite NCFI 24-124) foam was named NCFI 26-93 foam. The first lot of this foam was blended by LMC/MSFC at MSFC and shipped to MAF. MAF completed all planned sprays on this material, and the data that has been evaluated is quite favorable. Strength data on NCFI 26-93 foam appears to be comparable to that of NCFI 24-124 at a density of 0.1 lb/ft³ lower than that of NCFI 22-124.

13. The present plans call for using NCFI 24-124 on the LOX Tank and Intertank, and possibly NCFI 26-93 on the LH₂ tank. The first lot of NCFI 26-93 was blended at MSFC; Lot #2 was blended at NCFI. Although the 2nd Lot may have slightly more blowing agent than Lot #1, it apparently gives a foam that has a somewhat higher density. Part of the lower density of Lot #1 was due to the reduction of about 0.5 of a knit line in the sample. It was sprayed at 2 rpm and more foam per unit area, this giving a foam with knit line further apart. This gives a foam with more waviness (undesirable) than foam sprayed at 3 rpm and less foam per unit area. Waviness tends to increase as the duration of the spray increased. If NCFI 24-124 is used on the barrel section of the LH₂ tank, it will give a calculated weight increase of 146 lbs. of insulation above the base line [Base line foam NCFI 23-66(CFC-11 blowing agent)]. Present data shows that the use of NCFI 26-93 for the insulation will reduce the calculated weight increase to 101 lbs.

14. To try to reduce the density of NCFI 26-93 foam, 1% additional HCFC-141b blowing agent was added. Although no data was presented, it was orally stated that the foam was spongy and the appearance was poor.

15. Strength data on NCFI 24-124 foam and Lot #1 and Lot #2 of NCFI 26-93 foam all appear to be essentially the same, and meet requirements.

16. Recession rates on NCFI 26-93 run at Ames showed higher recession rates for this foam as compared to NCFI 24-124 foam; hence indicating the need for a greater thickness for this foam on the tank. This requirement for more foam on the LH₂ tank together with other minor effects reduced the calculated weight saving for using NCFI 26-93 on the LH₂ tank to 30 lbs. Based on this low potential weight savings, it was decided to stop work on NCFI 26-93 and to use NCFI 24-124 on the LH₂ tank.

B. SLWT WITH HIGHER OGIVE INTERNAL TEMPERATURE

1. SLWT has considered a possible requirement for a higher internal temperature allowable for the Ogive. The NCFI 24-124 foam for LWT on a back-face, temperature/vacuum test divoted around 320 to 330⁰F. It would not be expected that a ultralight NCFI 24-124 foam would divot at a much higher temperature. Tests on NCFI 24-57 foam (aft dome) divoted at 365⁰F just below the CPR-488 divoting range. Engineering has recommended to add more metal on the Ogive to keep the internal temperature to a maximum of 300⁰F.

2. It is surprising that NCFI 24-57 foam divoted at 10⁰F below the divoting temperature of CPR-488. NCFI 24-57 is a higher density and more thermally stable base material than the CPR-488 foam. The more thermally stable statement is based on TGA, thermal/vacuum, and windtunnel tests. The explanation for the lower divoting temperature of the NCFI 24-57 foam is due to a higher concentration of HCFC-141b being in the foam than that of CFC-11 in the CPR-488 foam. This higher concentration of HCFC-141b in the foam causes a higher pressure to form at the metal/foam interface when the metal is heated on the uninsulated side. If my theory is correct NCFI 22-65 (CFC-11 blowing agent) will probably not divot below 400⁰F.

3. In anticipation that it may be desirable at a later date to allow the internal temperature on the Ogive to rise to 350⁰ to 400⁰F, there are two foams that I know of that might be acceptable for use at these back-face temperatures. American Foam Technologies THERMO-COR™ new phenolic foam reportedly is available in sheet form or as a sprayable material in the 2 to 3 lbs/ft³ range that may have a closed-cell content exceeding 90% in some cases. It is reported that this foam can withstand temperatures up to 400⁰F without degradation. Another possible material of interest is ROHACELL 31WF and 51WF closed-cell rigid polymethacrylimide foam. These foams are reported to be dimensional stable up to 356⁰F. The 31WF foam has a density of 2.01 lbs/ft³ and that of 51WF foam a density of 3.21 lbs/ft

4. It is suggested that small samples of these foams be obtained for the purpose of running back-face temperature tests. It is proposed that these foams can be bonded to aluminum substrates with GX 6300 adhesive. Depending on the outcome of these tests and potential need, additional evaluation may or may not be warranted.

C. LOWER DENSITY SS-1171 FOAM FOR SLWT

1. There has been a minimum effort by LMC/MSFC to come up with a lower density SS-1171 foam. This has included among other things the addition of more HCFC-141b blowing agent and a change in catalyst. Tests on pours foams at LMC/MSFC showed that the addition of 1% more HCFC-141b to SS-1171 lowers the density of the foam about 0.1 lb/ft³. The addition of HCFC-141b to SS-1171 seems to have limited affect on the reactivity of the foam. The additions of Polycoat #5 catalyst to SS-1171 did not seem to have much effect on the density of knit lines or on the tendency of the foam when tested in tension to fail at knit lines. The SS-1171 foam may require a longer room temperature cure to lower the percentage of failures at knit lines when tested in tension. The addition of more blowing agent and cell modifying additions did not reduce the knit line density.

2. LMC/MSFC experience with BX-250 showed that lead based catalyst produced thicker and denser sprayed surfaces, while tin catalysts work synergistically with amines to produce thinner and lower density knitlines/sprayed surfaces. For the preliminary evaluation, LMC selected the most stable tin catalyst (T 125) and the most efficient blowing catalyst (Polycat 5) with the current amine catalyst (Polycat 8) for defining a catalyst package leaving out the current lead catalyst (lead naphthanate). They used bench work to select a catalyst package varying the three catalysts and catalyst levels. The catalyst package selected was based on reactivity, polyol compatibility, and foam discoloration. Only the best system was sprayed for testing. Originally the catalyst package selected was based on reaction speed and a smooth reactivity profile.

3. When foam was sprayed with the selected catalyst package, a pink tint was detected in the third and fourth spray passes. A new catalyst package was selected on minimum color change, reaction speed, and a smooth reactivity profile. Foam was also sprayed with this second catalyst package. Visual inspection of the sprayed foam indicated a reduced knitline thickness. Density of the two foams were 2.14 and 2.17 lbs/ft³ indicating that a lighter foam had been obtained. Mechanical tests were run on the two foams. The potential weight savings by using a lower density SS-1171 foam attracted little or no attention, hence efforts on this lower density foam system was dropped.

D. POSSIBILITY OF USING NCFI 24-124 ON THE AFT DOME LH₂ TANK

1. Since NCFI 24-124 and NCFI 24-57 foams have the same chemical materials, it was thought that there might be a possibility of insulating the aft dome of the LH₂ tank with NCFI 24-124 and obtaining a weight savings of about 50-70 lbs. The potential of this idea can be investigated rather cheaply. On this basis samples of NCFI 24-124, NCFI 24-57, NCFI 22-65, and CPR-488 foams were obtained for thermal/vacuum testing. Tests at 7 BTU/ft²-sec were run on specimens of 24-124 and specimens of 24-57 foams. The 24-57 foams definitely have a superior crack pattern as shown by approximately double the number of char islands with 24-57 foam than with 24-124 foam. Weight losses were about the same for both foams, and the 24-124 foam had a somewhat higher recession. One curious phenomenon encountered was the tendency of the char layer for the 24-124 foam to debond, and there may have been a trace tendency of the char from the 24-57 foam to also debond at the knit lines.

2. Based on the results of the 7 BTU/ft²-sec tests, it appears that 120 sec tests at 10 BTU/ft²-sec may be too long to have virgin foam left to get recession rates. A few tests at 10 BTU/ft²-sec for 120 sec showed that there was no virgin NCFI 24-124 foam left. Some tests were run for 80 sec., but most of the tests were run for only 60 sec. At these shorter times, there was virgin foam left for both the 24-124 and 24-57 foams. After considering all the thermal/vacuum test results, these tests indicated only 10 lb. weight saving by using NCFI 24-124 on the aft dome of the LH₂ tank. Based on these findings, no further consideration was given to using NCFI 24-124 foam on the aft dome of the LH₂ tank.

E. FOAM INSULATION ON SLWT

1. After all the testing on lighter weight foam materials, the potential weight saving by using these new materials did not justify solving all the new problems that their use would encounter. Hence there will be no new lighter weight foam insulation on the SLWT. Weight reduction in TPS will be limited to that obtained by stricter controls on the necessary TPS thickness in all areas.

V. ABLATOR MATERIAL ON THE ET LWT AND SLWT

A. SLA-561 PROBLEM ON LWT

1. A production operator noticed a metal particle embedded in a SLA panel near or on the surface. The metal particle proved to be stainless steel. A search showed that it came from a bearing from a mixer in which cork is processed. To the best of their ability LMC has concluded that about 25 grams of ground-up stainless steel could be in 700 lbs. of processed cork. SLA-561 made using this contaminated cork is on no less than 5 ET's and possibly one or two more. There is no concern for the damage these metal particles could do to Orbitor tiles. Unfortunately hits on the wind shield could be very damaging, and could come from metal contaminated SLA on the nose cone and upper few feet of the cable tray.

2. Although there is concern about possible damage to the wind shield of the orbitor from metal particles in the SLA, it is not believed that the metal particles will come off because there is little or no recession of the SLA during ascent. However, if tests are decided to be run, I suggest the SLA panel be fabricated with only metal particles embedded in the SLA surface of the uncured SLA panel in known locations. After hot gas or wind tunnel testing, tests could be made to see if the stainless steel particles are still on the tested SLA panels.

B. LOWER DENSITY SLA-561 FOR SLWT

1. There is still some 500 lbs. of SLA used on the ET. The SLA-561 can be made a lighter ablative material by decreasing the silicone resin content in the ablator. Considerable testing would be needed to qualify this lighter weight SLA for use on the SLWT. With the change to the composite nose cone, there will be considerable less SLA-561 on the ET, hence making the change to a lighter SLA even less attractive. In my opinion lighter weight SLA is not a good bet for weight saving.

VI. COMBINED ENVIRONMENT PANEL TESTING TO QUALIFY TPS MATERIALS FOR USE ON SLWT

A. COMBINED ENVIRONMENT PANEL FOR WELD TEST

1. The combined environment panel was fabricated of 2195 Al-Li metal with a weld in the center of the panel. The panel was so highly instrumented that this was not to be considered a TPS test. It had a 9" x 9" patch of hand packed SLA in the center of the test area. The whole test area had NCFI 24-124 foam as the insulation. In testing, failure occurred at 113% limit load while going to 125% limit load. The failure was in the parent Al-Li metal, and not in the weld. It was reported that no TPS failure occurred. The investigation of the failure concluded that a poor design was the cause of the failure.

B. COMBINED ENVIRONMENT PANEL SIMULATING TPS ON THE AFT DOME LH₂ TANK

1. The Combined Environment Panels was insulated with an area of bonded on SLA-561, NCFI 24-57 foam, and a close-out area over the SLA using SS-1171 foam. It was tested at Wyle and had successfully completed the ambient load calibration tests. The launch abort test had also apparently been successfully completed as loads on the X & Y axis's had been reduced to zero (from maximum of 396 & 342 KIPS Tensile) and the frame and control temperatures were at -80°F and -60°F. They were getting ready for the Launch Ascent Tests when the first pop was heard. Examination of the panel revealed TPS anomaly. After the 3rd pop, TPS and Metal failure was evident. Additional pops indicated progressive failures. Al-Li metal and TPS had come off the top of the panel, and apparently the SLA patch had failed cohesively. There were also cracks in the foam on the lower part of the panel.

2. The cause of the metal failure was probably due to compressive forces and torsional forces. Tests were run to try to determine what caused these loads to be applied when supposedly all tensile loads have been removed. The panel would be very susceptible to compressive loads, and it has been proposed that one of the load cells was applying tensile loads and another compressive loads. If Al-Li metal has an elastic limit, then compressive loads would have been introduced.

3. On stripping the TPS from the panel, there was no indication of primer or foam debond. The failure of the ablator was cohesive within the SLA-561. Also there was no indication of metal failure in the area of cracks in the TPS on the lower part of the panel.

C. WIDE PANEL TESTING

1. LMC/MAF proposed a new wide panel (wide cryoflex) test as a partial substitute for much more expensive combined environmental panel tests. MSFC was somewhat reluctant to buy into the new test without greater understanding of expected results. Before initiating this new test with the wide panels, pressure holes in the panel were eliminated which in my opinion was a good idea. Tests to date with the wide panels have been weld and structural tests. No TPS tests on wide panels are planned at this time.

VII. ET LWT COMPOSITE FAIRINGS

A. EFFECT OF INCREASED HEATING RATES ON COMPOSITE FAIRINGS

1. It was reported that hot gas testing of GR/Ep and GR/PI laminates encountered delamination in the first 3 layers of the composite (without encountering debris) when tested at the higher heating rates. Concern was expressed because of the delamination. Prior to testing, the laminates were exposed to high moisture for a minimum of 24 hours (95 to 100% relative humidity at 120°F and possibly higher). Moisture in composites acts as a plasticizer, softens the resin, and depresses the glass transition temperature. Hence moisture absorbed in the composite adversely effects high temperature performance.

2. Since moisture absorption is a slow process, I suggested they run hot gas tests on laminates exposed to high moisture for 24 hours, 72 hours and 168 hours to see if this would have any effect on the number of layers in a laminate that delaminated. I was told that this may have already been done.

3. While working for MSFC in the late 60's or early 70's, we ran some tests on moisture absorption/desorption in GR/Ep laminates. I also had a contract with General Dynamics studying the effect of moisture on composite. I found a couple of reports from General Dynamics on the subject in my files. I made copies of these reports, and distributed them to MSFC and LMC personnel to help them understand the moisture absorption problem.

4. GR/Ep Press Line Fairings are being flown based on Mission Specific. LMC back up position for composite fairings is to bond SLA on the fairing. They have run a few tests on bonding SLA-561 to PI composite. At ambient temperature the FWT was 45 psi; at 350°F FWT was 39 psi. Based on what I know about the procedure used, I am in agreement. Failure, as expected was in the SLA. I have not seen a plan for qualifying bonded on SLA.

5. I was asked to find out who the thermal/ablator man is at JSC. Through inquiries I was told that Steve Dorsey (713-483-6613) could tell you who he was.

VIII. WOODPECKER ATTACKED THE FOAM ON ET-71

A. While ET-71 was on the pad getting ready for flight early in June, 1995 woodpeckers attacked the foam on the ET over the Memorial Day weekend. They made holes in the foam all over the tank (nose cone, Ogive, barrel sections of LOX and LH₂ tanks, intertank, and LH₂ tank aft bulkhead). Since an early decision for roll-back to the VAB was not made, LMC/MAF furnished KSC emergency repair procedure that allowed PDL-4034 to be poured outside the accepted processing box. Twenty holes were repaired on the pad prior to the decision to roll-back to the VAB. By the time of flight in July all the repairs to the foam were adequately cured.

B. Considerable testing was done on partially cured PDL-4034 pour foam. These included hot gas test, thermal/vacuum test, back face temperature/vacuum test, tensile test, etc. No unusual popcorning or divoting occurred during hot gas or thermal/vacuum tests, and everything considered none of the tests data indicated that PDL-4034 foam was insufficiently cured after 48 hours or longer to justify postponing flight.

C. Woodpecker repellents are being considered.

1. Southwest Research Institute has a patent on using isophorone as a repellent. This material is somewhat toxic and may be a carcinogen. The usual range of application is between 0.5 oz. to 3.0 oz. per ft². However if encapsulated it can be applied as low as 0.1 oz. per ft². It may only be effective for 5 to 6 weeks, hence it would need to be applied just after or just prior to roll-out. It is not recommended to be tested because of its toxicity, its limited time of effectiveness, the amount of repellent required, and because of its general lack of field application.
2. A much better bet for a woodpecker repellent is a Lockheed/DuPont material formed in the production of saccharin. It is supposed to be effective for 2 to 3 years and will require much less material (probably around 25-40 lbs. per ET). It is understood that LMC/MAF plans to investigate this material.

IX. PROBLEMS WITH THE PRIMER USED ON THE ET

A. PROBLEM ENCOUNTERED WITH CHANGE IN OWNERSHIP AND MOVE OF PRODUCTION FACILITIES

1. DeSoto, the producer of the 515-346 primer used on the ET, was purchased by Courtaulds Aerospace, Inc. Not only were the two men that had blended the primer for LMC changed, but the production facility was moved from Berkeley to Mojave facility. This move of facility was dictated by California laws.
2. The first lot of primer from the Mojave facility did not meet all the RAP tests-wt. per gallon and also the salt fog test. The second lot passed all the RAP tests. LMC has a third lot of primer on order. LMC has enough primer manufactured at Berkeley to last until September 1996. There is no data on primer blended at the Mojave facility on 2195 Al-Li. The primer that will be applied to the first SLWT will be from the Mojave facility.

B. FUTURE POTENTIAL PROBLEMS WITH THE ET PRIMER

1. There is considerable pressure being exerted to discontinue the use of a hexavalent chromium as the corrosion inhibitor in all primer systems. To my knowledge none of the new proposed corrosion inhibitors are anything like as effective as the chromate inhibitor. I do not know when the chromate inhibitor may be removed from the primer systems.

2. There is also a move to eliminate resin systems in primers that require objectionable solvents. Courtaulds Aerospace, Inc. is planning on going to water soluble resin systems in their primer. On this basis they are presently planning on discontinuing the production of Super Korapon resins (used in the ET primer) during the first quarter of 1998. Courtaulds has promised LMC to continue small lot production of Super Korapon resin as needed for the ET primer. Product quality on those small lots of resin may not be the same as on large lots of resin (more variability), hence LMC will probably have to increase RAP testing as a minimum, to insure that the consistency of the resin system has not changed. In my opinion, I do not believe that primer based on water soluble resins will meet ET temperature requirements - flexibility at -423°F , adequate strength at $+300-350^{\circ}\text{F}$.

C. SUGGESTED POSSIBLE REPLACEMENTS FOR THE ET PRIMER

1. Based on the somewhat uncertainty of the continued availability of the present ET primer system, it seems desirable to find out if the thermal plastic polyimide resin can be thermally sprayed on aluminum without adversely affecting the temper of the 2219-T87 aluminum. If the thermal plastic polyimide resin can be thermally sprayed on aluminum without adversely affecting the temper of the metal, then I think it would be advisable to try to incorporate in the thermal spray one or more of the more promising corrosion inhibitors that appear to be candidates as replacement for the chromate corrosion inhibitor.

2a. Should the thermal sprayed polyimide coating be successful, it would still be desirable to have available a primer in solution for repairs such as scratches and also for small components. Although the primer would undoubtedly have undesirable solvents, exceptions could possibly be obtained due to small amounts used. The present epoxy based primer or its alternate with a new corrosion inhibitor could be used for this application.

2b. Another approach for finding a replacement for the present ET epoxy primer would be to use the thermal plastic polyimide resin as the base resin in a new primer paint system. This turned out to be a poor idea as we have not found a solvent for the thermal plastic polyimide resin (Meldin 3000 F from Furon Advanced Polymers Division). The Meldin 3000 F resin showed little or no solubility in methylene chloride. Trying to use any advanced high-temperature thermoplastic resin in a paint system encounters the problem of finding a solvent as these materials as a whole are noted for their chemical resistance.

2c. We had a supposedly thermal plastic polyimide resin from Ciba-Geigy that is soluble in several solvents to at least 20%. This resin was dissolved in a combination of three solvents, and a coating on aluminum was made using a doctor blade. After an extended cure at ambient temperature, tests on the strength of the coating to the aluminum showed failure at the aluminum/coating interface at around 260 psi (undesirably low). Later we learned that the resin we tested is actually a soluble polyimide precursor that after application can be converted to a thermoset polyimide by an extended cure of several hours at 350-500°F. Strength properties are greatly improved by conversion to the polyimide, but these high cure temperatures will destroy the temper of 2219-T87 aluminum.

3. Another possibility would be to use the modified epoxy resin system that is the base resin in Lefkowied 109/LM-52 adhesive. The company manufacturing Lefkowied 109/LM-52 adhesive was taken over by Teck Form Co. which was later taken over by Kester Salder Division of Litton Systems, Inc. On the Apollo program Lefkowied 109/LM-52 and perhaps Lefkowied 211 adhesions were used at -423°F. Lefkowied 109/LM-52, as I remember, was not too strong at temperatures above 200°F, and Lefkowied 211 had better high temperature properties. I have no idea on the availability of these resin systems or how easily they could be used in paint systems.

X. THERMAL SPRAY APPLICATION OF POLYMER COATINGS

A. During my efforts in 1993 and 1994, it was suggested that thermally sprayed polymer coating on metals could possibly replace the present primer used on the ET and also on some parts of the SSME. If successful as a corrosion inhibitor, this thermally sprayed coating would eliminate the problem with volatile organic chemicals (VOC), and also the potential problem with hexavalent chromium, a potential carcinogen. Prior to completing the earlier study, purchase orders were placed for a thermoset cyanate ester, a thermoplastic polyimide resin, and a thermoplastic polyetherimide.

B. Progress on this project was limited while I was not available. I found, upon return, only one of these polymers had been received for this project. Some effort had been made to spray this thermoplastic polyimide polymer by Rocketdyne with very little success. The other two resins have since been received. The polyetherimide resin from GE is in pellet form. Rocketdyne shipped this material to New Jersey where it was made into a powder by hammer milling. The cyanate ester (PT-60) from Allied Signal arrived in 3 pails in a single solid form. This material is quite brittle, being easily broken and ground to a powder. This powder tends to set back-up in about 2 to 18 hours. However, when mixed with titanium dioxide (TiO_2) ~10/90 or 20/80 ratio of TiO_2 to polymer, the powder remains fluid.

C. Processing problems have been encountered with the cyanate ester (PT-60). In an attempt to ballmill this resin, the powder resin stuck to the sides of the ballmill, and except for the outer layer it appeared to be the same as the unball-milled resin. This tendency of the powder, even mixed with TiO_2 , to stick back together hinders the screening of the resin. A small sample of the screened resin with approximately 23% titanium dioxide (TiO_2), although fairly stable powder, would not flow properly when an attempt was made to thermally spray the mixed resin. Silicone dioxide (SiO_2) is reported to be more effective in keeping the resin in the powder state, but this has not been tried.

D. Rocketdyne sent about 50 pounds of the polyetherimide resin (in pellet form) to Allgrind Plastics in New Jersey to be ground to a 50 to 70 micron particle size. Allgrind was unsuccessful in attempting to ballmill this polyetherimide resin. They then borrowed a hammermill, and reduced the size of the pellets. Allgrind encountered some trouble with a screen. The end result is that there is 22 lbs. of contaminated material (with screen), about 20 lbs. of material -100 to -200 mesh, and about 10 lbs. of material -200 mesh (desired).

E. Rocketdyne signed a contract with Applied Polymer Systems, Inc. to use their Thermoplastic Plasma Spray Process to coat aluminum and stainless steel samples with each of the three resin materials that we had purchased. They did not want to start processing until we furnished them both the 3 resins and also the desired aluminum and stainless steel substrate specimens to coat.

F. The Weidman Company, Inc. uses a High Velocity, Oxygen Fuel (HVOF) Plastic Spray System to apply resin coatings on metal and other substrate. They offered to attempt to spray our polyimide and polyetherimide resins. They had trouble with the polyimide resins because the powder size was too small and with the polyetherimide because the powder size was too large. They need a powder size in the range of 50 to 70 microns. When they mixed our resins with HDPE, they were able to obtain coatings on steel that were about 1.5 mil thick and that appeared to have good adhesion to the steel. They have offered to spray two of our resins for an affordable price.

G. In an effort to increase our supply of the desired size powder for thermal spraying, we ballmilled some of the 70+ micron size polyetherimide powder. This resin was ballmilled for approximately 20 hours. Part of this ballmilled resin was placed on a screen and the screen was shaken on a mechanical shaker for 3 1/2 hours. Only very limited quantity of the ballmilled resin went through the 75 micron screen. Essentially all of the powder that went through the screen stayed on the 53 micron screen. Based on these results it was concluded that ballmilling of the already powder resin was not an attractive way to get the 50 to 70 micron powder resin that is needed.

H. Personnel from Rocketdyne visited the Weidman Company at Fort Myers, Florida. Weidman demonstrated the HVOF system by spraying two polymers on 2219 aluminum substrates. Neither the 50-70 micron powder polyetherimide or the blend of polyetherimide and polyimide resins produced coatings that had good adhesion to the aluminum substrate. The deposited coatings appeared to be about 2 to 3 mils thick.

I. Rocketdyne also visited Applied Polymer Systems (APS) at Tampa, Florida. They use a modified plasma thermal spray to apply resins on metal substrates. APS was under contract to Rocketdyne to spray 3 resins on both 2219-T87 aluminum and 21-6-9-Cres.

J. APS had trouble spraying the polyetherimide resin, and seemed to think the size of the powder was too large although it was a 50-70 micron powder. The sprayed resin had a rough surface. The dark coating that was obtained may indicate thermal decomposition or at least a color change that occurs above 720°F. We do not know whether argon or high pressure air was used in processing. If air was used in processing, a dark color coating would not be unexpected. Coating thickness as measured by APS ranged from 4 to 8 mils.

K. The polyimide resin powder ranged in size from 13 to 50 microns. The coatings as deposited were dark in color and rather rough. Coatings thickness varied from 2 to 6 mils. Better coatings may have been obtained if the size of the powder had been larger.

L. The coating with the best appearance was obtained with the cyanate ester. This resin requires special processing. Cold resin is ground in a blender, and the powdered resin has to be returned to the freezer. If left at room temperature for 12 hours or longer, the powder will flow back together and will have to be reground. When the gun is ready to spray, the cold resin is placed in the feed hopper of the gun. The coatings were applied in thickness of 3 to 12 mils.

M. Before any testing is done on the cryogenic bond samples, thickness measurements will be made to be sure we know the actual thickness of the coatings. We plan on running bond adhesion tests on the coatings. Since the coating thicknesses exceed the thicknesses we wanted (0.5 to 1.5 mils), corrosion tests probably will not be run.

N. Rocketdyne is planning on a contract with The Weidman Company to thermally spray 2 or 3 resin systems for us. They have obtained some of Aurum Thermoplastic Polyimide resin, and we have some polyetherimide resin on hand. Some information from Allied Signal indicated that if 0.5 to 1.0% of silica is added to powdered cyanate ester that it will prevent the powder from coagulating back together. On this basis we put 200g of cold cyanate ester in a cold blender with about 1.5g of silica. After about 4.5 minutes of grinding at low speed, the blender stopped due to odor. There was some real fine powder on the side of the blender, and some reduce size pellets (powder plus fine gravel size resin) in the bottom above some caramelized resin. The heat generated by the grinder heated the resin too hot. Less than 5 hours later the resin that was not caramelized would no longer pour. It was believed that most of the silica was in the caramelized resin.

O. On the above assumption some of the cyanate ester was ground in mortar and pestle into a somewhat coarse powder and placed in a clear jar. To this 1.2% silica was added to the semi-powder resin and was mixed by shaking. In less than 30 minutes it stuck together to some extent and in 2 hours it was pretty difficult to break-up. Based on these results, the cyanate ester has been deleted from the thermal spray program until a better method is developed for keeping the ground resin in a powder state.

P. The Aurum Thermoplastic Polyimide resin has a particle size mostly in the 40 to 60 micron range. The Weidman Company thermally sprayed some of this resin, and got a good coating on metal about 4 mil thick. Unfortunately, there was some humps in the coating probably due to only partially melted 100 micron powder. It was reported that the coating stuck quite well to the metal substrate, and they believe that they can spray this material to a 2 mil or less thick coating. Later inspection of the coated samples showed that the coating was not sticking adequately.

Q. Rocketdyne ran some bend test at room temperature on samples that were prepared by Applied Polymer Systems. All coatings were thicker than desired (3-10 mils thick vs. desired of max of 2 mils). Only samples applied to stainless steel were tested. The cyanate ester coating cracked when the specimen was bent around a one inch mandrel. When a tape test was run on the bent area, the tape removed the cyanate ester coating. Although the cyanate ester coating looks good, it does not have the properties that are needed for space application. The coating of polyetherimide resin appeared as if it may have been burned and the surface of the coating had a rough sandpaper like finish. This coating was tested over a one inch, a three quarter-inch, and one half-inch mandrels. When a tape test was run on the bent area, some outer particles on the coating was removed. No cracks in the coating were detected visually. The polyimide coating performed better than the other two coatings and passed all 3 mandrel bend tests.

R. Rocketdyne ran some additional bend tests at around -300°F that were thermally sprayed by Applied Polymer Systems. The polyimide coating on stainless steel was bent around 1-inch, 1/2-inch, and 1/4-inch mandrels. There were no indications of cracking or lift-off of the coating, and tape test on the bent area did not remove any of the coating. The polyimide coating on aluminum was only tested on 1/2-inch and 1/4-inch mandrels. Again, there were no indications of cracking in the coating or loss of any coating by the tape test. The polyetherimide coating on aluminum was only tested on the 1/4-inch mandrel. Here there was some indication of cracks in the coating and some loss of burned material by the tape test. Tests on the polyetherimide coating will be discontinued until a better coating is obtained.

S. Rocketdyne reported that the 2219-T87 aluminum on which Applied Polymer System had thermally sprayed a polyimide coating, hardness had dropped from 73-75 to about 20. Unfortunately we do not know the temperature history of the sample (how hot they were and how long at the elevated temperature).

T. Aluminum samples coated with the 3 resins were placed in a salt spray cabinet. After 15 days, the burnt sandpaper-like coating of polyetherimide resin had general corrosion thus showing the porosity of this coating. The polyimide coated specimen showed good protection for the metal except in scribed areas on the panel, after 30 days in the cabinet.

U. Portable adhesion test results were run on the polyimide coating. All breaks occurred at the adhesive/coating interface at between 1666 psi and 2196 psi (10 specimens), hence the coating adhesion to the metal is above 2196 psi.

V. The Weidman Company used their HVOF Plastic Spray System to apply coatings of polyimide, polyetherimide, and a mixture of polyimide/Peak resins to metal samples. The coatings on all the specimens looked rough and maybe partially burned, and appeared as if they could be removed easily. However, one specimen passed a 1/4" bend test at room temperature. In my opinion HVOF process is not the way to apply the resin coatings.

W. Plasma Processes, Inc., Tim McKechnie, showed a sample of a thermoplastic polyimide resin that he had thermally sprayed on stainless steel. The coating looked good and appeared to have good adhesion to the metal. Coating thickness appeared to be about 2 to 3 mils. Based on the appearance of the sample, he was asked to come up with a plan on what he might do to meet ET requirements. Later I contacted him about the status of his proposal. I was told he had been unable to find a place to get 12 feet of 12" diameter 2219-T87 pipe. I talked to LMC about the possibility of them making the pipe. Later Tim found a source for the 2219-T87 pipe.

X. At my suggestion, Tim submitted two cost proposals for thermally spraying the polyimide coating: one for him to furnish the 12 feet of 12" pipe and the other for MSFC furnishing the 12 feet of pipe. The difference was about \$6,000, and I believe that LMC could fabricate the 12 feet of pipe cheaper than that. I like the proposal in fact that they will try to control and measure the temperature of the aluminum during the thermal spray. At present no funding has been found for this effort.

XI. THIRD GENERATION BLOWING AGENTS

A. HCFC-141b PHASEOUT OPTIONS IN 1-1-2003

1. High costs are expected for development, validation and implementation for a new blowing agent for ET foams by 2003. Present regulations allow the use of HCFC-141b past 2003, but importing and manufacturing of the blowing agent are forbidden. Stockpiling of 141b is not too attractive an option for a number of reasons. NASA may ask EPA for a waiver on one or both of the options for importing or manufacturing of 141b to avoid the high cost of qualifying a new blowing agent. For either of the two options to work for NASA other markets must be available for HCFC-141b. At this time there are other major industries considering the waiver option.

B. THIRD GENERATION BLOWING AGENTS

1. LMC/MSFC has been looking at the following potential blowing agents: HFC_s, HFE_s, FIC_s, and H₂O. Their efforts have been hampered by the high cost of the blowing agents HFC_s, HFE_s, AND FIC_s. None of these are commercially available. Water has been successfully used in pour foams, but has not been as successful in spray foam. More success has been obtained by using H₂O along with HFC_s or HFE_s in spray foams. All efforts to date have been with SS-1171 (a polyurethane foam), PDL-1034 pour foam (a polyurethane foam), and in-house polyurethane pour foams.

2. Three HFC and two HFE have been successfully used as blowing agents in the SS-1171 formulation. Using a high pressure Gusmen processing unit, higher densities foams were obtained with the HFC material that had boiling points below the 25°C to 30°C (desired blowing agent boiling range). It was shown that the foam densities obtained with HFC 236 ea (B.P. 6.5°C) processed with low pressure Edge-Sweets equipment gave lower density foams as compared to the same foams sprayed with the high pressure Gusmer equipment (2.4 vs. 2.9 lb/ft³). The HFC_s used were all fluorinated propanes. No fluorinated butanes have been obtained as yet. Blowing agents with boiling points above 30°C encounter a different set of problems like those encountered with HCFC-141b (B.P. 32°C) when it was substituted for CFC-11. Only solubility data has been obtained with these new blowing agents in polyisocyanate foams, and there appears to be no problem with solubility.

3. It is reported that HFC 245 fa (B.P. 15.3°C) will be commercially available from Allied Signal. Solvay and Elf Atochem plan to commercialize HFC 365 (B.P. 24.9°C). Reports indicate that HFC 365 has limited solubility in foam components.

4. It is believed that the HFC will not be as soluble in the sprayed foam as either CFC-11 or HCFC-141b. If the HFC has a boiling point below 25°C , there will be a more rapid rate of escape from the cells with air replacement resulting in a higher thermal conductivity for the foam at an earlier date. Where HCFC-141b replaced CFC-11 in NCFI 22-65 foam for the aft dome of the LH_2 tank, the strain capability of the new foam (NCFI 24-57) was improved over that of the NCFI 22-65 foam. If and when HFC blowing agents are used in the NCFI foams replacing HCFC-141b, there probably will be a decrease in the strain capabilities of the new NCFI foams. On the positive side lower concentrations of blowing agent retained in the foam could improve the foam resistance to higher internal temperatures such as might be encountered on the Ogive.

5. Another potential group of blowing agent is based on FIC. These materials as blowing agents give me concerns. These blowing agents would need to be kept exceedingly dry because I would expect the iodine molecule will be easily hydrolyzed off yielding HI gas and a potential chain stopper.

XII. AEROGELS FOR INSULATION ON CRYOGENIC TANKS

1. Aerogels are molecular-scale open-cell foams that are made using supercritical processing (over 1000 psi pressure at 300°C or higher). Processing condition and time required are reduced by using liquid CO_2 supercritical drying. Aerogels have a high porosity, but passage of air and gelling solvents through aerogels are extremely slow due to the close packing of molecular-scale particles. Densities of the aerogels range from 0.05 to 0.44 g/cm^3 .

2. Silica aerogels are made from methyl or ethyl ortho silicates in an alcoholic medium. The thickness of aerogel panels are usually limited to approximately 3cm thick because of the time required to leach out gelling solutions. Silica aerogels are not very strong. Using Hardman A-85 polyurethane adhesive they got a bond strength to a substrate with 0.38 g/cm^3 (about 23 lbs/ft^3) aerogel of about 15 psi; with 0.15 g/cm^3 aerogel a bond strength of 2.5 psi. With silica aerogel there would be no problem of oxidation in an elevated temperature environment.

3. Organic aerogels are made by the aqueous polycondensation of (1) resorcinol with formaldehyde and (2) melamine with formaldehyde. Organic aerogel precursor can be cast to near net shape and will suffer a uniform linear shrinkage of about 25% during pyrolysis. Carbon aerogels at a density of 0.1 g/cm^3 has a surface yield strength of about 30 psi and can be elastically strained up to several percent. Carbon aerogels would have to be protected from oxidation in an elevated temperature environment.

4. Before aerogels can be used on flight vehicles, it would need to be tested to be sure that the panels would not destruct from rapid evacuation. I do not believe the aerogel could be bonded directly on a LH_2 tank because I believe that cryopumping would occur, and when the LH_2 level fell below the cryopumped air, the change from liquid to gaseous air would blow the aerogel off the tank. I presume that aerogel panels will have to be made in desired shape and sizes, and that they cannot be thermally or otherwise formed to desired shape. I have no information on how large aerogel panels can be made, and if the size increases, can cracking be prevented?

5. Based on the information I have, the most attractive use for aerogel insulation would be on reusable flight vehicles (one stage to orbit). This is based on the assumption that a refractory coating can be applied over the aerogel that will allow the aerogel to survive both ascent and re-entry heating. If this is the case, then the total weight of insulation on a LH_2 tank would be around 1.0 to 1.5 lbs/ft^2 . The break-down of insulation weight follows:

Approx. lbs/ft^2	Material
0.2	1.0 inches of 2.4 lb/ft^3 polyisocyanate foam (0.5 inch could be sufficient).
0.1	Adhesive to bond aerogel to foam (would also cover wt. of primer on tank).
0.7	Assuming 3 cm. layer of aerogel, with a density of 0.1 g/cm^3 (about 6 lbs/ft^3), and a 100 micron refractory coating on the aerogel with a density of 5.
----- 1.0	Total

Thermal diffusion time constant for a 3 cm. thick layer of carbon aerogel is approximately one hour. Since coated aerogel could oxidize at joints or small leaks, it may be desirable to increase the thickness of refractory coating to attempt to eliminate these potential oxidization areas and to enhance the coatings ability to resist erosion from ascent and re-entry. It might also be desirable to enhance the strength of the carbon aerogel by increasing the density. These increases could increase the total weight of the TPS to approximately 1.75 lbs/ft².

6. The potential of aerogels for insulation for a single stage to orbit flight vehicle is sufficient for at best NASA keeping abreast of the aerogel development. As resources become available, I think the following tests should be considered:

- a. A small sample of sealed carbon aerogel (with refractory coating if possible) should be exposed to rapid evacuation equivalent to that experience in ascent. This is possibly not necessary, but is such a simple, inexpensive test that it should be done to enhance safety and confidence.
- b. Testing for cryopumping is not necessary as long as there is a layer of foam insulation between the aerogel and the LH₂ tank wall. I believe the foam insulation layer is necessary to eliminate all possibilities of cryopumping occurring.
- c. I propose to bond the aerogel to the foam insulation with a silicone adhesive such as RTV-560 or GX6300, and also the aerogel panels to each other.
- d. There is probably some effort needed in learning to apply the refractory coating to the aerogel.
- e. A best effort possible is needed to calculate how hot the adhesive bond line between the foam and the aerogel may get during re-entry.
- f. Tests are needed to see how well this insulation concept will stand-up to aero-shear and aero-heating during re-entry, and also radiant heating simulating aft dome conditions during ascent.

Items 6a and 6e should be done as soon as feasible because information developed in these tests will be influential in determining how to proceed. Only minimum funding will be required for these two tests, and item 6e is the most critical. The aerogel panel used in item 6a could possibly be used to develop information needed for item 6e. Item 6e should be updated periodically as more information becomes available.

7. If the bond line temperature between the foam and the aerogel is much above 300°F, then it would be necessary to consider a foam with higher temperature capabilities such as ROHACELL or phenolic foams.

8. Silica aerogels would be preferred over organic aerogels when strength properties are increased. This preference is based on no requirement to protect the aerogel from oxidation in an elevated temperature environment.

XIII. LINER FOR GRAPHITE/EPOXY COMPOSITE LOX TANK

A. In an effort to save structural weight on a one component flight vehicle capable of going into orbit and return to earth with minimum refurbishment required between flight, designers are looking at using graphite/epoxy composite to fabricate LOX tanks. Some people are saying that certain graphite/epoxy composites are LOX compatible. It has also been reported that LCP and polyimides are also LOX compatible. All three of these resins, epoxy, LCP and polyimides, have hydrogen atoms along the polymer chains that would be free to react with LOX/GOX, and hence, I am concerned if these materials are used in LOX tanks.

B. Drexel University and Foster-Miller, Inc. reported that the thermally sprayed FEP coating they applied to graphite/epoxy composite had very low bond strength. The sample of coating I saw looked like the FEP resin had not really stuck to itself and hence it did not stick very well to the graphite/epoxy surface. I expressed a concern that FEP Teflon coating applied to a composite would possibly encounter cracking of the coating due to differences in thermal expansion/contraction when subjected to LOX temperatures.

C. MSFC Nonmetallic Materials Branch is considering a contract with a qualified vendor to thermally spray a FEP Teflon coating on graphite/epoxy. Data could be obtained on bond strength and also on the ability to withstand cryogenic temperatures.

D. Earlier we had an idea of using a 1 mil Kapton H/0.5 mil FEP Teflon Film for a LOX composite tank liner. We showed that a bond could be formed by heat between a piece of Kapton H film and the FEP Teflon side of the composite film. The interesting feature of the Kapton H/FEP Teflon film was that the film could not be straightened out, but curled into a roll. To me this indicated the FEP resin had a greater thermal contraction than the Kapton film; hence, my concern with cracking of a thermally applied FEP coating on graphite/epoxy when exposed to cryogenic temperatures.

E. If this idea is given more consideration, I believe the next film should be 1 mil Kapton H/1 mil FEP film (more bonding film). To fully evaluate the idea I think a hand held heated roller is needed capable of temperatures of at least 700°F.

XIV. MISCELLANEOUS

A. SPRAY PHENOLIC FOAM

1. We were visited by a representative from American Foam Technology. They supposedly have a sprayable phenolic foam. Georgia Pacific makes the phenolic resin prepolymer. The company used a different prepolymer for every 5 pound change in density of the foam such as 1-5 lb., 5-10 lb., etc. The desired density of the foams are obtained by changes in the catalysts. To obtain 6 month storage life, the phenolic resin prepolymer is stored at 50-60°F. The catalyst ("B" component) can be stored at room temperature. Flexible phenolic foams are made by varying the catalyst. At present a 3 lb. flexible phenolic foam is made by using 2 catalysts as the "B" & "C" components and substrate needs to be 50°C or higher (not above 70°C) to keep the foam from slumping. They have used a resorcinol adhesive for bonding the phenolic foam. The foams are blown by CO₂. TGA data shows that weight loss is initiated at around 450°F. For a 2 to 2.5 lb/ft³ density foam the close cell content should be around 87-90%.

2. Months later, American Foam Technologies brought about 10 gallons of their Spray Phenolic Foam to MSFC. This foam was blended to give about a 2 to 4 lb/ft³ foam. Initial efforts to spray the phenolic resin was done at ambient temperature conditions and a 20-30 sec. overlap time. Considerable slumping occurred and some debonding of the foam at the knit lines also occurred. Somewhat better foam was obtained when the substrate and the components were heated. Foam sprayed on the vertical surfaces had densities ~2.5 lbs/ft³, and flatwise tensile strengths at room temperature of 7.5 to 10 psi.

3. Horizontal sprays were also made. No back face heating was possible with the horizontal sprays. Overlap times of 4-5 seconds was used. With this technique a 14 lb/ft³ density foam was obtained with no obvious knit line. On heating, knit lines did appear. The flatwise tensile strength for this foam at -320°F, 75°F, 200°F, and 300°F were 16, 39, 20, and 10 psi, respectively. For this density, the strength of the foam is low.

4. Both the 2.5 lb/ft³ foam and 14 lb/ft³ foam absorb water showing a high open-cell content. This problem might be taken care of by applying a silicone coating, preferably a methyl phenyl silicone coating. The foam does not catch on fire easily, but once lighted it tends to keep on smoldering after the flame is taken away. I was also told that foam continues to cross link on standing. If the foam cross links for long periods of time, it would become more brittle; hence affecting the foam capability to perform satisfactorily. On this basis considerable improvement in this foam system will be needed before it can be considered as a possible replacement for spray SLA-561.

B. USBI SPRAY SYSTEM THAT USED NO ADDED SOLVENT

1. USBI ABLATOR MCC-1 THAT IS APPLIED BY SPRAY WITH NO SOLVENT

a. USBI developed a spray system for applying the ablator MSA to the solid motor that does not use a solvent in the application. This modified ablator is called MCC-1. LMC/MAF is very interested in coming up with a replacement for their spray SLA-561 primarily due to an environmental problem associated with disposing of the heptane that is used in the spray process.

b. At the request of LMC/MAF, a telecon was arranged involving MSFC, USBI and LMC/MAF. During the telecon it was learned that the system on the Center is primarily a production unit that is being used to qualify MCC-1 for use on the Titan, and that any investigative work should be done with the unit off the Center. USBI has made ablator coating with less resin than used in MCC-1. MCC-1 has a density around 35 lbs/ft³. It is quite hard and probably brittle, and I would not expect it would be usable at cryogenic temperatures. LMC requested samples of the MCC-1. I suggested that LMC get USBI to try to spray a somewhat similar-like material to SLA by using SLA ingredients except substituting a different resin for the silicone resins in SLA. Later, it was learned that USBI had already used silicone resins in their spray equipment.

- c. At my last contact, lawyers have reached an agreement protecting USBI's proprietary rights so that USBI can try to spray SLA-561 using their solventless spray system.

C. FOAMS FOR USE ON THE COMPOSITE NOSE CONE TEST

LMC has selected a silicone foam as a filler for the gap that will occur between the composite nose cone and the foam on the Ogive when the tank is filled with LOX. For the application a low density methyl phenyl silicone foam would be better than a dimethyl silicone foam because of its lower glass transition temperature T_g . If the temperature gets below -150°F then polyimide foam would be better since it is already below its T_g at ambient temperature. I furnished them with some information on a one lb/ft³ closed cell polyimide foam.

D. AXAF SOLAR ARRAY POTENTIAL PROBLEM

Testing of the SOLAR ARRAY PANELS is being done at -200°C (-328°F), and may be encountering debonds of the solar cells. Based on past experience the fabricator of solar panels do not like to use a primer to bond the solar cells to the substrate because of the potential of repairs being necessary. For two part silicon adhesives as a whole only limited bond strength is obtained without the use of a primer.

E. PREFLIGHT STRONG CAUSTIC SPILL ON STS-75

1. CAUSTIC SPILL BY USBI

- a. In a cleaning operation USBI spilled some 50 gallons of a strong caustic solution that may have encountered the TPS on the ET. The TPS was washed down with water for some 3 to 4 hours. Based on my experience with foam, it was my opinion that no serious damage had been done to the ET TPS by this caustic spill. Both MSFC and USBI ran some tests trying to duplicate the spill, and concluded that no major damage had been done. STS-75 took off as scheduled, and no TPS problems on the ET were reported.

XV. CONCLUSIONS AND RECOMMENDATIONS

1. All four foams containing the more environmentally friendly blowing agent HCFC-141b that are used on ET LWT have been qualified and successfully applied on flight hardware.
2. The light weight versions of NCFI 24-124 and SS-1171 have been evaluated for possible use on the ET SLWT. Potential weight savings were not attractive enough to continue qualification efforts.
3. None of the new foams if used on the Ogive would allow the internal temperature of the Ogive to be raised to 400°F, and 350°F would be questionable. Phenolic foam and polymethacrylimide foams are suggested as potential foams for use on the Ogive if 350° or 400°F internal temperatures are required.
4. A new method for selecting foams to be qualified for back-up for ET LWT was suggested. This is based on working with the foam vendor to replace materials in his foam formulation that are either more critical or ones that appear to have a least stable market.
5. The future of the present ET primer has been made somewhat uncertain due to change in environmental constraints. Thermal spray of a thermal plastic polyimide resin has been suggested as a potential alternate. A potential source of a modified epoxy resin has been located, and based on past performance, it should have a good potential for use in a primer system replacing the Super Koropon resin used in the present ET primer.
6. Thermally sprayed coatings on metal surface should be useful for corrosion protection. Test have shown that the thermal spray of a thermal plastic polyimide resin gave the best coating of the three resins that were thermally sprayed. If the thermal plastic polyimide resin can be thermally sprayed on aluminum without adversely affecting the temper of the aluminum, then the polyimide coating has an excellent possibility of meeting ET requirement over a temperature range of -423°F to +400°F. Assuming the thermally sprayed polyimide coating is successful, considerations should be given to incorporating in the spray one or two of the most promising corrosion inhibitors that show promise for replacing the chromate corrosion inhibitor in the present ET primer.

7. LMC has used possible third generation blowing agents (HFC and HFE - fluorinated substituted propanes and fluorinated substituted methyl ethyl ethers) in pour foams and in a polyurethane spray foam and tests show that they have adequate solubility in isocyanate foam components. From available information HFC based on fluorinated butanes may not be adequately soluble in foam components. I expect the HFC will be less soluble in processed foams, and the strain capability of these foams will be less than that of foams blown with CFC and HCFC.

8. Aerogels are open cell foams and would not be expected to be usable as an insulation on liquid hydrogen tanks due to cryopumping. Aerogels applied over polyisocyanate foams may be a way to protect the cryogenic foam from high temperatures encountered in ascent and re-entry provided the aerogel can be adequately protected by a refractory coating. Silica aerogels would be preferred over carbon aerogels provided that adequate strength properties can be developed. Aerogels may have use as a cryogenic insulation on LOX tanks.

9. Thermally sprayed coating of FEP Teflon has been suggested as a liner for composite (resin/graphite composite) LOX tank. I am concerned because of the potential of FEP Teflon coating to crack at LO₂ temperature from differences in coefficient of contraction.

10. It was suggested that a coating for a LOX composite tank could be made from a DuPont film of Kapton H/FEP Teflon film. To fully evaluate the potential of this suggestion a hand held heated roller is needed with 700°F capability. Differences in coefficient of expansion could also effect the usability of a liner of this type.